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Letter Report

Fabrication of Intermetallic Compounds by
Solid State Reaction of Roll-Bonded Materials

Contract No. N00014-85-C-0857

Prepared for:

Office of Naval Research
Arlington, VA 22219

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Quarterly Progress Report (#16)
(MCR-85-721)

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1.0 **Contract Number:**
N00014-85-C-0857

2.0 **Reporting Period:**
5/10/90-8/9/90

3.0 **ONR Scientific Officer:**
Dr. Steven G. Fishman

4.0 **Work Performed At:**
Martin Marietta Astronautics Group, Denver, CO 80201

5.0 **Principal Investigator:**
Dr. William C. Moshier

6.0 **Project Title:**
Fabrication of Intermetallic Compounds by Solid State Reaction of Roll-Bonded Materials

7.0 **Description of Research:**

7.1 **Objectives of Present Research**

- Intermetallic compounds offer very high specific material properties and property retention at elevated temperatures. However, reliable processing techniques to fabricate these materials have not been developed. This program will investigate the use of a novel, low cost fabrication technique involving a deformation-solid state reaction bonding process to form titanium beryllides. The objectives of this investigation are to:
 - Establish reaction kinetics for the formation of Ti beryllide intermetallic compounds,
 - Establish processing parameters needed to reproducibly fabricate these materials,
 - Characterize the properties of Ti beryllides fabricated using a deformation-solid state reaction bonding process,
 - Investigate the effects of alloying additions on the crystal structure of Ti beryllides and determine how these changes impact mechanical properties.

7.2 **Summary of Work Accomplished During Previous Reporting Period**

- Characterization and Testing of Ti Beryllide Foils

Determining the composition and structure of the beryllide sputter deposited foils was in the process of being performed during the last quarter. The results of the analysis indicated that the foils contained no detectable oxygen using AES. The crystal structure of the foils made during the last quarter, all of which had excess Be over the composition needed for $TiBe_{12}$, was $TiBe_{12}$, indicating that the Be may be substituting with Ti in the crystal.

7.3 Progress During the Reporting Period

- Room Temperature Testing of Ti Beryllide Foils

During this period, the foil fabrication effort was completed. Table 1 shows the range of compositions as determined by WDS. The composition range, between 90 and 94.6 a/o Be, was characterized using x-ray diffraction to determine the crystal structure of the material tested. The results of this analysis show that the crystal structure that formed during deposition onto the substrate Mo-coated Cu substrate is $TiBe_{12}$. Figure 1 shows the diffraction spectra for the foils at 90.05 and 94.62 a/o beryllium.

Preliminary analysis of the foil crystal structure at high Be concentrations (94 a/o) using transmission electron microscopy indicates that the material is indeed $TiBe_{12}$. However, the intermetallic contains a very high density of growth and misfit dislocations that arise during the sputtering of the intermetallic onto the Mo-coated Cu substrate. There does not appear to be any oxide particles or other second phase particles in the material.

The tensile fracture strength of the foils was measured over the range of Be concentrations. The general trend is that the tensile strength of the beryllide increases with Be concentration, as shown in Figure 2. The fracture surface on all of the samples is smooth at low magnifications. At higher magnifications, the failure is transgranular and the features on the fracture surface change significantly with Be concentration, as shown in Figure 3. The surface appears smoother and more cleavage-like as the Be concentration decreases. This change in fracture morphology correlates with the decrease in fracture strength. There are isolated areas on several Be-rich samples that appear to be crystallographic in nature, as can be seen in Figure 4.

- Auger Sensitivity Factor Evaluation

A study was performed to accurately quantify the AES results obtained on this program over the past year. This study involved using the sputtered beryllide foils as a standard, and determining their concentration with WDS. The WDS data was correlated to the AES peak-to-peak information, and this data was used to determine the functional dependence Be/Ti peak-to-peak data to the atomic concentration of Be in the sample. The functional dependence is shown in Figure 5. Figure 6 illustrates the degree by which the Be concentration obtained from the sensitivity factors determined on pure metals underpredicts the Be concentration obtained by WDS.

Using the functional dependence developed on this program, we now know from the original AES that the $TiBe_{12}$ phase is present in the reaction zone in the co-extruded samples. Subsequent diffraction data confirms that $TiBe_{12}$ is present in the reaction zone closest to the Be fibers in the first co-extrusion. The small difference in the Be concentration between $TiBe_{12}$ and Ti_2Be_{17} make it difficult to distinguish between these compounds in the SEM.

7.4 Tasks for the Next Period

- Interim Report

At this time, we have completed the first phase of this program, which involved defining the processing conditions for fabricating Ti beryllides using a solid state reaction process. An Interim report will be compiled and delivered to ONR near the end of August and will document the work that has been accomplished in the first phase of this program.

- Reaction of Ti-Be Laminate

The laminate sheet that was rolled down to 0.026-in.-thick has been cut into several sections, including two 3" x 3" sections that have been recanned for rolling. This procedure was necessary to reduce the laminate spacing so that the sheet could be reacted in a limited six hour HIP cycle. The rolling schedule has been identified so that the final thickness of the laminate will be 0.008-in.-thick. After this sheet has been reacted, the mechanical properties of the beryllide will be determined, and compared to the sputter deposited beryllide foil. We anticipate having monolithic beryllide sheet/foil ready for testing by the end of the next quarter.

- TEM Analysis of Foils

A heat treatment study will be conducted to determine the temperature required to anneal out the growth dislocations. The structure of the material that was grown by sputtering is complex, and addition work will be required to understand the preliminary TEM results obtained at the end of the this reporting period.

- High Temperature Tensile Testing of the Beryllide Foils

High temperature tensile tests will be performed on the foil samples in air at 1200°C (note: the testing temperature stated in the last letter, i.e., 2000°C, was a mistake).

7.5 Presentations During This Period

- None

7.6 Technical Reports During This Period

- None

7.7 Publications During This Period

- None

7.8 Participants On The Program (Last Quarter)

Name	Task
Nuclear Metals Inc., Concord, MA.....	Fabricate MCF/Extrusion/Specimen Prep
EG&G, Rocky Flats, Golden, CO.....	Sputter-deposit Be/ Ti-Be films
Manufacturing Sciences Corp, Oak Ridge, TN.....	Can Material, Performing Roll Bonding
LANL, Los Alamos, NM.....	TEM of Ti beryllide foils

Table 1. Ti and Be Concentrations and Foil Thickness of the Sputter Foils

Run Number*	WDS Concentration (a/o)				Thickness by weight (μm)
	Ti	±σ	Be	±σ	
6	6.78	0.006	93.21	0.006	50.3
7	6.76	0.013	93.23	0.013	50.0
8	6.49	0.011	93.50	0.011	44.2
9	5.37	0.023	94.62	0.023	45.2
10	6.02	0.003	93.97	0.003	47.3
11	9.94	0.006	90.05	0.006	57.6
12	8.65	0.021	91.34	0.021	55.9
13	8.17	0.005	91.82	0.005	54.6
14	7.77	0.003	92.22	0.003	54.0

* Runs 1-5 were used to standardize the process

a)

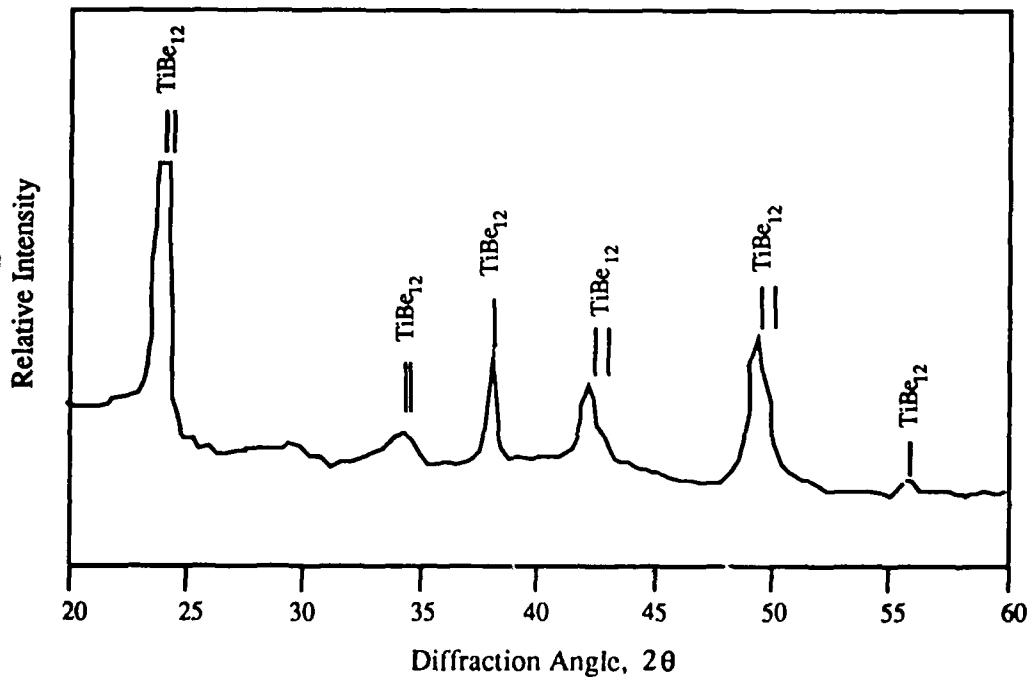
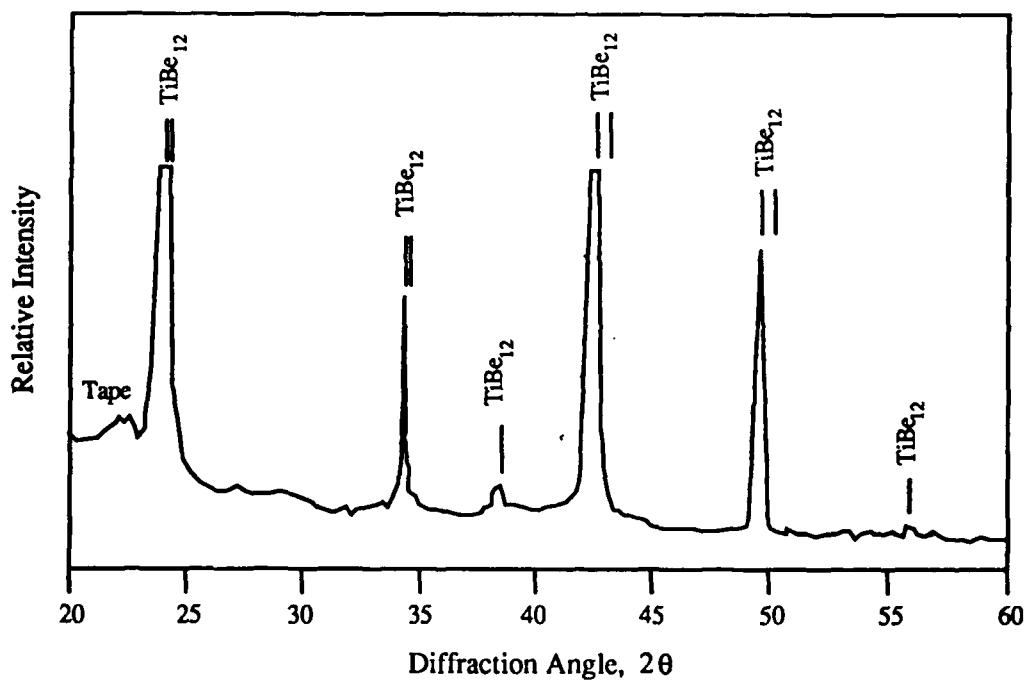


Figure 1. X-ray diffraction spectra for Ti-Be co-sputtered foils at 500°C for a) 90.05 at/o Be and 94.62 at/o Be.

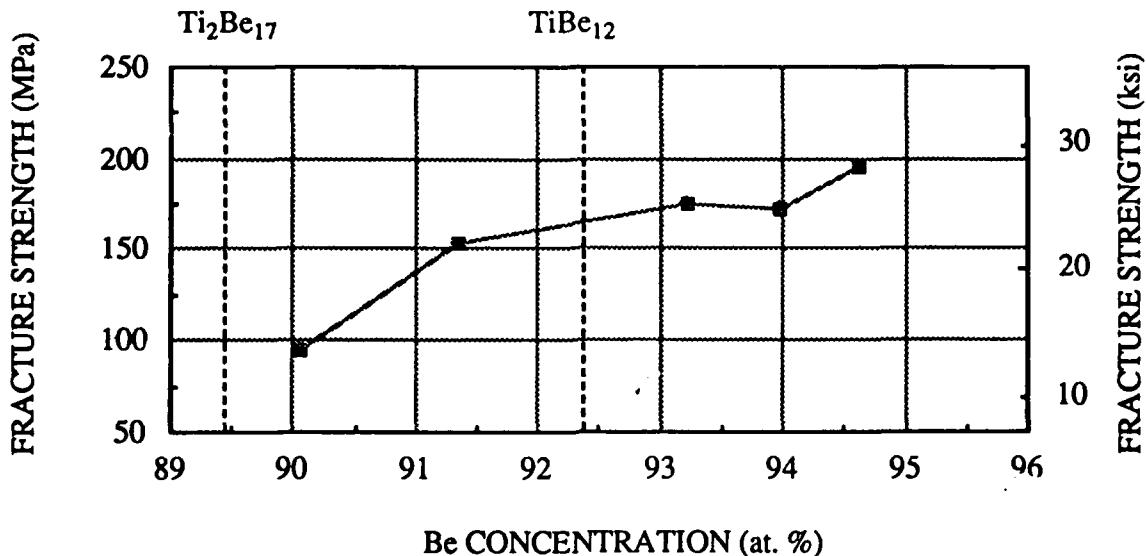


Figure 1. Fracture strength as a function of Be concentration indicates that the strength of the beryllide increases with Be concentration. Note that the actual structure at each Be concentration is actual $TiBe_{12}$.

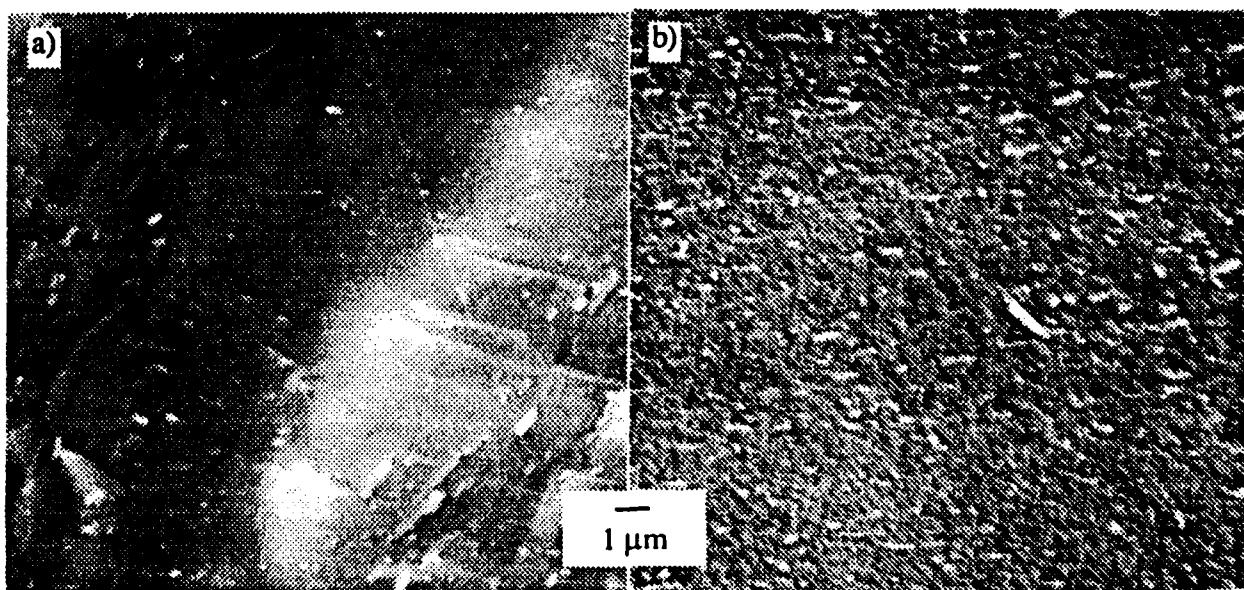


Figure 3. Fracture surface of Ti beryllide foil containing a) 90.05 a/o Be and b) 93.97 a/o Be. The smooth cleavage-type fracture of the sample containing 90.05 a/o Be corresponds with a lower fracture strength.

a)



b)

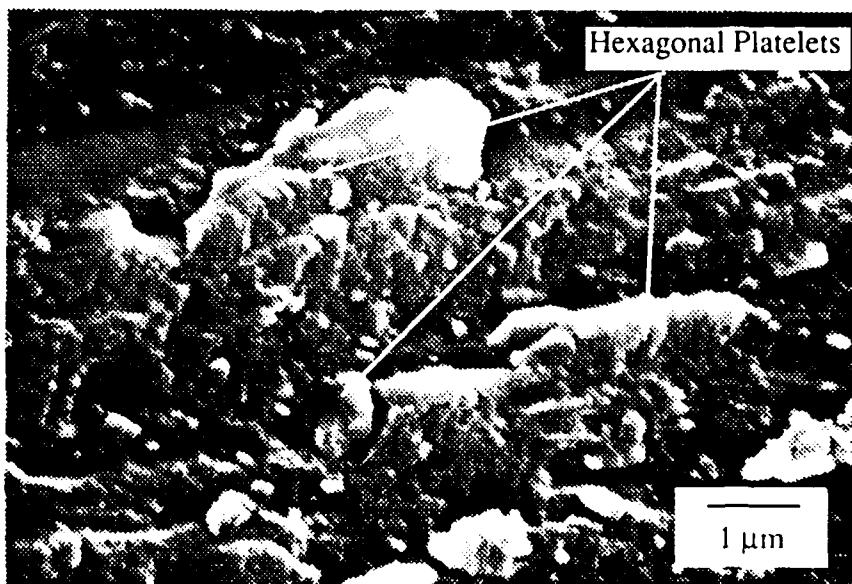


Figure 4. Isolated area on sample containing 93.23 a/o Be showing: a) rough areas banded by smooth regions, and b) microstructure of rough region, which contain hexagonally-shaped platelets.

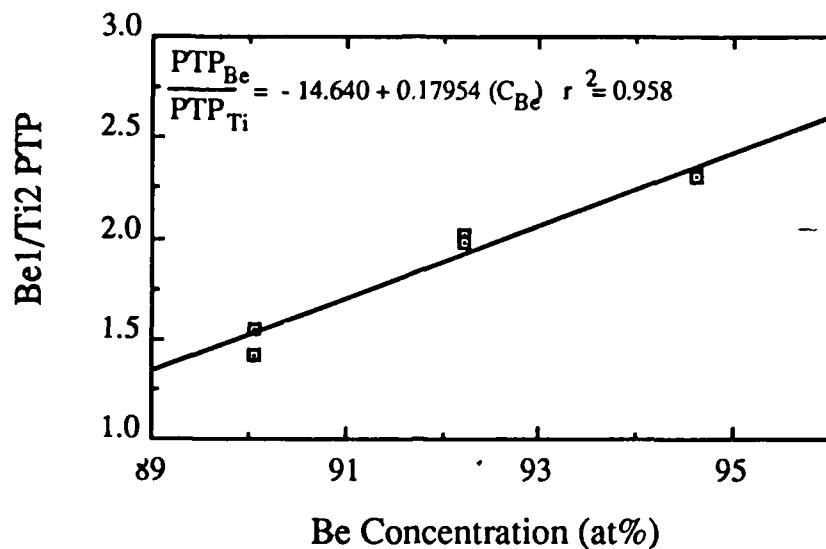


Figure 5. Correlation between the ratio of Be to Ti Auger peak-to-peak (PTP) data plotted against the atomic concentration of Be in the foil as determined by WDS (C_{Be}).

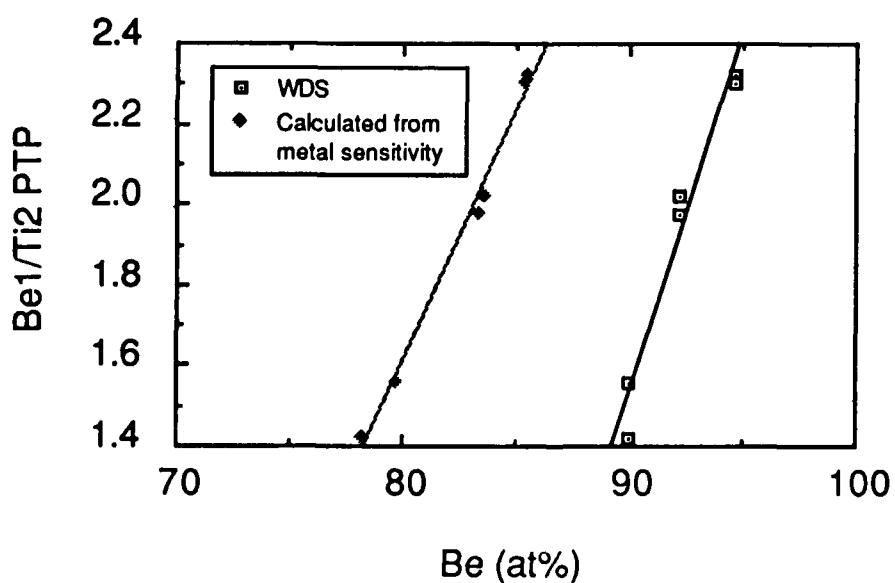


Figure 6. A comparison of the Be/Ti PTP versus WDS determined Be concentration shown in Figure 5, and the calculated concentration using the Be/Ti PTP data and the sensitivity factors measured on the pure metals.